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Date of Filing : 24 FEBRUARY 2001

Application Number : 200100727-7

Applicant(s) /  
Proprietor(s) of  
Patent : URAH RESOURCES (NIGERIA) LTD;  
OLOBO JONATHAN OBAJE

Title of Invention : SOLVENT-FREE TRANS-ACIDOLYSIS  
PROCESS FOR THE PREPARATION OF  
EDIBLE SURFACE-ACTIVE  
CARBOHYDRATE FATTY-ACID ESTERS

  
SHARMAINE WU (Ms)  
Assistant Registrar  
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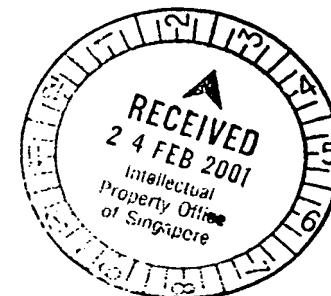


24 FEB 2001  
200100727-7

Rule 19

SINGAPORE  
PATENTS ACT  
(CHAPTER 221)  
PATENTS RULESThe Registrar of Patents  
Registry of PatentsREQUEST FOR THE GRANT OF A PATENT  
THE GRANT OF A PATENT IS REQUESTED BY THE UNDERSIGNED ON THE BASIS OF THE PRESENT APPLICATION

<i>I. Title of Invention</i>		SOLVENT-FREE TRANS-ACIDOLYSIS PROCESS FOR THE PREPARATION OF EDIBLE SURFACE- ACTIVE CARBOHYDRATE FATTY-ACID ESTERS	
<i>II. Applicant(s)</i> <i>(See note 2)</i>	<i>(a) Name</i>	Olobo Jonathan Obaje Tel (65) 7744432	
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<i>Country</i>			
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<i>Body Description/ Residency</i>			
<i>State</i>			
<i>Country</i>			

#160  
24/2  
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<i>III. Declaration of Priority (see note 3)</i>	<i>Country/Country Designated</i>		<i>File no.</i>	
	<i>Filing Date</i>			
	<i>Country/Country Designated</i>		<i>File no.</i>	
<i>IV. Inventors (See note 4)</i>				
(a) <i>The applicant(s) is/are the sole/joint inventor(s).</i>	<input checked="" type="checkbox"/>	<i>Yes</i>	<input type="checkbox"/>	<i>No</i>
(b) <i>A statement on Patents Form 8 is/will be furnished.</i>				
<i>V. Name of Agent (if any) (See note 5)</i>				
<i>VI. Address for Service (See note 6)</i>	<i>Block No</i>	510	<i>Level No</i>	04
	<i>Unit No</i>	311	<i>Postal Code</i>	120510
	<i>Street Name</i>	West	Coast	Drive
	<i>Building Name</i>	Blk 510		
<i>VII. Claiming an earlier filing date under section 20(3), 26(6) or 47(4). (See note 7)</i>	<i>Application No</i>			
	<i>Filing Date</i>			
	<i>[Please tick in the relevant space provided]:</i>			
<input type="checkbox"/> <i>Proceeding under rule 27(1)(a).</i> <i>Date on which the earlier application was amended =</i> _____ <i>or</i> <input type="checkbox"/> <i>Proceeding under rule 27(1)(b).</i>				

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VIII. Invention has been displayed at an International Exhibition (See note 8)		<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No																											
IX. Section 114 requirements (See note 9)		The invention relates to and/or used a micro-organism deposited for the purposes of disclosure in accordance with section 114 with a depositary authority under the Budapest Treaty.																												
		<input type="checkbox"/> Yes	<input type="checkbox"/> No																											
X. Check List (To be filled in by applicant or agent)		<table border="1"> <tr> <td colspan="2">A. The application contains the following number of sheet(s):-</td> </tr> <tr> <td>1. Request</td> <td>4</td> <td>sheets</td> </tr> <tr> <td>2. Description</td> <td>11</td> <td>sheets</td> </tr> <tr> <td>3. Claim(s).</td> <td>2</td> <td>sheets</td> </tr> <tr> <td>4. Drawing(s).</td> <td>—</td> <td>sheets</td> </tr> <tr> <td>5. Abstract.</td> <td>1</td> <td>sheets</td> </tr> </table> <table border="1"> <tr> <td colspan="2">B. The application as filed is accompanied by:-</td> </tr> <tr> <td>1. Priority document</td> <td></td> </tr> <tr> <td>2. Translation of priority document</td> <td></td> </tr> <tr> <td>3. Statement of Inventorship &amp; right to grant</td> <td></td> </tr> <tr> <td>4. International Exhibition Certificate</td> <td></td> </tr> </table>		A. The application contains the following number of sheet(s):-		1. Request	4	sheets	2. Description	11	sheets	3. Claim(s).	2	sheets	4. Drawing(s).	—	sheets	5. Abstract.	1	sheets	B. The application as filed is accompanied by:-		1. Priority document		2. Translation of priority document		3. Statement of Inventorship & right to grant		4. International Exhibition Certificate	
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XI. Signature(s) (See note 10)		<table border="1"> <tr> <td>Applicant (a)</td> <td>OLDBO JONATHAN OBATE <i>Anthony</i></td> </tr> <tr> <td>Date</td> <td>24 Feb 2001</td> </tr> <tr> <td>Applicant (b)</td> <td></td> </tr> <tr> <td>Date</td> <td></td> </tr> <tr> <td>Applicant (c)</td> <td></td> </tr> <tr> <td>Date</td> <td></td> </tr> </table>		Applicant (a)	OLDBO JONATHAN OBATE <i>Anthony</i>	Date	24 Feb 2001	Applicant (b)		Date		Applicant (c)		Date																
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Date	24 Feb 2001																													
Applicant (b)																														
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ENCLOSED US DISCLOSURE DOCUMENT NO 475021  
 Dated JUNE 01 2000.

NOTES:

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1. This form when completed, should be brought or sent to the Registry of Patents together with the prescribed fee and 3 copies of the description of the invention, and of any drawings.
2. Enter the name and address of each applicant in the spaces provided at paragraph II. Names of individuals should be indicated in full and the surname or family name should be underlined. The names of all partners in a firm must be given in full. The place of residence of each individual should also be furnished in the space provided. Bodies corporate should be designated by their corporate name and country of incorporation and, where appropriate, the state of incorporation within that country should be entered where provided. Where more than 3 applicants are to be named, the names and address of the fourth and any further applicants should be given on a separate sheet attached to this form together with the signature of each of these further applicants.
3. The declaration of priority at paragraph III should state the date of the previous filing, the country in which it was made, and indicate the file number, if available. Where the application relied upon in an International Application or a regional patent application e.g. European patent application, one of the countries designated in that application [being one falling under the Patents (Convention Countries) Order] should be identified and the name of that country should be entered in the space provided.
4. Where the applicant or applicants is/are the sole inventor or the joint inventors, paragraph IV should be completed by marking the 'YES' Box in the declaration (a) and the 'NO' Box in the alternative statement (b). Where this is not the case, the 'NO' Box in declaration (a) should be marked and a statement will be required to be filed on Patents Form 8.
5. If the applicant has appointed an agent to act on his behalf, the agent's name should be indicated in the spaces available at paragraph V.
6. An address for service in Singapore to which all documents may be sent must be stated at paragraph VI. It is recommended that a telephone number be provided if an agent is not appointed.
7. When an application is made by virtue of section 20(3), 26(6) or 47(4), the appropriate section should be identified at paragraph VII and the number of the earlier application or any patent granted thereon identified. Applicants proceeding under section 26(6) should identify which provision in rule 27 they are proceeding under. If the applicants are proceeding under rule 27(1)(a), they should also indicate the date on which the earlier application was amended.
8. Where the applicant wishes an earlier disclosure of the invention by him at an International Exhibition to be disregarded in accordance with section 14(4)(c), then the 'YES' Box at paragraph VIII should be marked. Otherwise the 'NO' Box should be marked.
9. Where in disclosing the invention the application refers to one or more micro-organisms deposited with a depository authority under the Budapest Treaty, then the 'YES' Box at paragraph IX should be marked. Otherwise the 'NO' Box should be marked.
10. Attention is drawn to rules 90 and 105 of the Patent Rules. Where there are more than 3 applicants, see also Note 2 above.
11. Applicants resident in Singapore are reminded that if the Registry of Patents considers that an application contains information the publication of which might be prejudicial to the defence of Singapore or the safety of the public, it may prohibit or restrict its publication or communication. Any person resident in Singapore and wishing to apply for patent protection in other countries must first obtain permission from the Singapore Registry of Patents unless they have already applied for a patent for the same invention in Singapore. In the latter case, no application should be made overseas until at least 2 months after the application has been filed in Singapore.

## For Official Use

Application Filing Date: / /

Request received on : / /

Fee received on : / /

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\*Delete whichever is inapplicable

## TITLE OF THE INVENTION

SOLVENT-FREE TRANS-ACIDOLYSIS PROCESS FOR THE  
PREPARATION OF EDIBLE SURFACE-ACTIVE CARBOHYDRATE  
FATTY-ACID ESTERS

5

## BACKGROUND OF THE INVENTION

## (a) Field of the Invention

The present invention relates to process for production of edible carbohydrate fatty-acid esters, and in particular, a low temperature, solvent-free trans-acidolysis reaction between carbohydrate ester of low molecular-weight carboxylic acid and free fatty-acid under reduced pressure and in the present of an acid catalyst.

## 15 (b) Description of the Prior Art

Carbohydrate fatty-acid esters are odorless, non-irritant nonionic surfactants with excellent biodegradability properties. They are used as low calorie fat substitute, or to solubilize membrane proteins, and 20 to formulate many grades of detergents, pharmaceutical, food and cosmetic products. Carbohydrate fatty-acid esters also exhibit anti-microbial and insecticidal activities and are used as cerebral metabolism improving agent, and in the treatment of colonic disorders. But the synthetic methods for carbohydrates fatty-acid 25 esters are faced with many limitations.

U.S. Patent No. 5,945,519 describes a process for the solvent-free preparation of sucrose fatty acid esters and their mixtures with



nonsugar polyol fatty acid esters. In this process, a sucrose is reacted with one or more fatty acid alkyl esters of a chain length of 6 to 20 carbon atoms and the reaction mixture is then reacted at reduced pressure with fatty acid alkyl ester, and then filtered without addition of solvent.

5 U.S. Patent No. 4,614,718 discloses the synthesis of sugar or sugar-alcohol fatty acid esters by incubating an aqueous mixture of a sugar or sugar-alcohol, a C8 – C22 fatty acid and a catalytically 10 active amount of a lipolytic enzyme derived from the genus *Pseudomonus*, *Enterobacterium*, *Chromobacterium*, *Mucor* or *Candida* at a temperature from about 20 degree C to about 60 degree C, and recovering the resulting ester from the mixture.

15 15 U.S. Patent No. 5,908,922 (issued to Kasseri, et al, June 1, 1999) discloses a method for producing a sucrose fatty acid ester, which comprises subjecting sucrose and a lower alkyl ester of fatty acid to an ester exchanger reaction in an organic solvent in the presence of an alkali catalyst to produce the corresponding sucrose fatty acid 20 ester.

25 U.S. Patent No. 4,927,920 (issued to Wagner, et al, 22 May 1990) discloses a method for making sugar esters by reacting a sugar and an ester of the sugar having a degree of substitution greater than about 2 in an organic solvent in the presence of a transesterification catalyst to form a sugar ester having a degree of substitution up to 2;



and recovering the sugar ester having a degree of substitution up to 2 at a temperature below the boiling point of the organic solvent.

U.S. Patent No. 4,996,309 discloses a process for preparing 5 sucrose fatty acid esters by reacting sucrose and a fatty acid alkyl ester in an aqueous reaction system in the presence of a catalyst, adding water to the reaction mixture to dissolve it, adjusting the reaction mixture to a neutral pH region, adding a neutral salt to the solution to precipitate the sucrose fatty acid esters, separating and 10 washing the precipitate with an acidic water, and subjecting the washing liquid to ultrafiltration.

The conventional methods for producing carbohydrate fatty esters are encountered with the following disadvantages and limitations:

15 (1) the transesterification method, currently favoured by most industrial producers of carbohydrate fatty esters, is a reversible process with poor conversion equilibria because the substrate sugars and fatty acid moieties are immiscible due to polarity differences. Thus, low product yield 20 characterize the processes;

(2) Mutual solvents, such as pyridine, N, N-dimethylformamide (DMF), dimethylacetamide (DMA), dimethylsulfoxide (DMSO), chloroform, benzene and toluene, are required to solubilize the substrates. These mutual solvents are toxic 25 and cannot be removed to the level compatible with current regulations, thus limiting product applications.



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- (3) High process cost resulting from high-energy cost and complex purification procedures.
- (4) Thermal degradation and isomerization of the reactant carbohydrates at temperatures above 100 degree C, which is normally required in most known processes.

10 It is a principal object of the present invention to provide a solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters, which obviates the above drawbacks and/or limitations.

#### SUMMARY OF THE INVENTION

15 Accordingly, it is an object of the present invention to provide a solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters, wherein the process comprises a low temperature, solvent-free trans-acidolysis reaction between carbohydrate ester of low molecular-weight carboxylic acid (C2 to C3) and free fatty acid (C6 to C22) under reduced pressure and in the 20 presence of an acid catalyst.

25 Yet another object of the present invention is to provide a solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters, wherein mono-, di- and poly-fatty esters of C2 and C3 acylated carbohydrates, with hydrophile-lipophile balance (HLB) values of 2 to 10, are obtained depending on the initial molar ratio of the reactants and chain-length of the fatty acid moiety.



A further object of the present invention is to provide a solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters, wherein acid or base catalyzed partial deacylation lead to carbohydrate fatty esters with free hydroxyl groups, and having HLB values of 8-18.

Yet a further object of the present invention is to provide a solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters, wherein the unreacted free fatty acids and carbohydrate esters of low molecular-weight carboxylic acids, which are removed during purification and recycled to the starting reactant mixture.

The advantages of the process in accordance with the present invention, which circumvent the above limitations, are

- (1) avoiding solvent as a reaction medium. The C2-C3 acyl group attached to the reactant carbohydrate is a good protecting and leaving group and, at the same time, enhances the solubility of the carbohydrate moiety in the fatty acid;
- (2) using low pressure to drive the reaction equilibrium forward at a reduced energy demand. This results in high yield (>90), less isomerization and degradation of products.
- (3) The feedstock is renewable natural resources, readily available and cheap. Unreacted substrates are recycled. This process is thus commercially viable.



(4) The invention is applicable to both batch-wise carbohydrate fatty ester synthesis and to continuous synthesis.

5 (5) High-grade (98% pure) carbohydrate fatty acid esters can be obtained.

(6) This process is adaptable for producing a number carbohydrate fatty acid esters.

10 Other objects and advantages of the invention will become apparent upon reading the following detailed description.

#### DETAILED DESCRIPTION OF THE PRESENT INVENTION

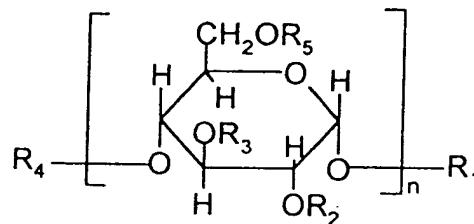
15 The present invention will be described in detail.

In accordance with the solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters of the present invention, acylated carbohydrate with chain-length C2-C3 includes mono to trisaccharide carbohydrates [see molecular 20 structure], and the free fatty acid has chain-length C6 – C22.

In the present process, the acid catalyst includes concentrated sulphuric acid and alky sulfonic acid polysiloxanes. The carbohydrate fatty-acid esters of the present process has the following molecular structures:

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wherein R2, R3, R5 are each selected from the group consisting of H, CH<sub>3</sub>CO, CH<sub>3</sub>CH<sub>2</sub>CO, and C6-C22 fatty acyl group;

5 R1, and R4 are each selected from the group consisting of a partially or peracylated mono- or di-saccharide, H, CH<sub>3</sub>CO, CH<sub>3</sub>CH<sub>2</sub>CO or C6-C22 fatty acyl group; and n is an integer with value equal to 1, 2, or 3; wherein the primary monosaccharide unit, i.e. the [---]n unit, could be a furanosyl, pyranosyl or C2-C6 open-chain structure.

10

In this process, acylated (C2 to C3) carbohydrate, preferably, mono to tri-saccharide) is reacted with free fatty acid (C6 to C22) and the reaction temperature ranges from 60 to 95 degree C, preferably from 80 to 90 degree C, in the presence of an acid catalyst, under

15 reduced pressure within the range of 4 to 20 Torr, preferably from to 5 to 10 Torr and, without adding any solvent. Next, the reaction mixture is decolorized and separated into unreacted fatty acid layer and a carbohydrate fatty-ester layer.

The unreacted C2-C3 acylated carbohydrate is then

20 precipitated out by cooling the carbohydrate fatty-ester layer to 10 to 0 degree C. If desirable, the free hydroxyl groups are librated by partial hydrolysis in the presence of an acid or base catalyst. The unreacted free fatty acids and carbohydrate esters of low molecular-



weight carboxylic acids, which are removed during purification, are then reused. The low molecular weight carboxylic acid, which is a by-product of the trans-acidolysis, is trapped and recycled.

5 In accordance with the preferred embodiment of the present invention, the process is carried out at low temperature, within the range of 60 to 95 degree C.

10 The following examples will further illustrate the present invention.

**EXAMPLE 1: Preparation of acetylated glucose laurate and the partially deacetylated glucose laurate**

0.03 mol (6.00g) of lauric acid (Fluka Chemika, Switzerland) was 15 added into a three-necked, round-bottom flask equipped with a magnetic stirrer, stopcocks, a vacuum take-off line leading to a liquid nitrogen cold-trap and a vacuum pump. 0.01 mol (3.90g) of Glucose pentaacetate, GPA, (Fluka Chemika, Switzerland) was added and the mixture was heated to 80-100°C in an oil bath with continuous stirring until a homogenous solution 20 was formed. 0.1 % w/w (0.01g) of concentrated sulphuric acid was then added and 10-20 Torr pressure was applied. The reaction was continued for 3 to 6 hours. The reaction was then stopped and taken up with 100ml of n-propanol. The solution was then neutralized with 5.0ml aqueous 1M



NaHCO<sub>3</sub>, decolourised with 1.0g activated charcoal and filtered warm. (Ethyl acetate or acetonitrile could also be used in place of n-propanol). N-propanol was then removed by vacuum distillation.

Unreacted lauric acid was extracted with 50 X 4 ml hexane. Hexane 5 was vacuum distilled to recover unreacted lauric acid and kept for reuse. The glucose fatty ester was redissolved in warm n-propanol, cool to 0 °C and filtered to give unreacted GPA as precipitate. The filtrate n-Propanol was then vacuum distilled off to give 70-85% pure acetylated glucose laurate. Repeated fatty acid extraction and GPA precipitation gave 90-95% pure 10 product (90.2% yield).

#### **Partial deacetylation of acetylated glucose laurate**

Acetylated glucose laurate obtained above was divided into four equal portions. Each portion was refluxed in 150ml 1MHCl (aqueous) solution for various lengths of time, to give partially deacetylated glucose fatty esters of 15 various HLB values, depending on the length of reflux. The solvent was distilled off under reduced pressure.



**Example 2: Preparation of acetylated sucrose oleate and the partially deacetylated sucrose oleate**

0.06mol (16.93g) of oleic acid (Fluka Chemika, Switzerland) was weighed into a three-necked, round-bottom flask equipped with a magnetic stirrer, stopcocks, a vacuum take-off line leading to a liquid nitrogen cold-trap and a vacuum pump. 0.01mol (6.79g) of sucrose octaacetate, SOA, (Fluka Chemika, Switzerland) was added and the mixture was heated to 80-100°C in an oil bath with continuous stirring until a homogenous solution was formed. 0.1 % w/w (0.024g) of concentrated sulphuric acid (or 0.5g alkyl sulfonic acid polysiloxane) was then added and 10-20 Torr pressure was applied. The reaction was continued for 3 to 6 hours. The product was taken up in 250ml of ethyl acetate, neutralized with 5.0ml aqueous 1M NaHCO<sub>3</sub>, decolourised with 2.0g activated charcoal, and filtered. Ethyl acetate was then removed by vacuum distillation.

Unreacted oleic acid was extracted with 50x4 ml hexane and kept for reuse. The sucrose fatty ester was redissolved in warm n-propanol, cool to 0 °C and filtered to give unreacted GPA as precipitate. The filtrate n-Propanol was then distilled off to give 70-85% pure acetylated sucrose oleate. Repeated hexane extraction and SOA precipitation gave 90-95% pure product (90.2% yield).



### Partial deacetylation of acetylated sucrose oleate

Acetylated sucrose oleate obtained above was divided into four equal portions. 150 ml aqueous ammonia solution was added to of the portions and 5 agitated for various times, to give sucrose fatty esters of various HLB values, depending on the length of treatment with the ammonia solution. The solvent was distilled off under reduced pressure.

While the invention has been described with respect to 10 preferred embodiment, it will be clear to those skilled in the art that modifications and improvements may be made to the invention without departing from the spirit and scope of the invention. Therefore, the invention is not to be limited by the specific illustrative embodiment, but only by the scope of the appended claims.

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## CLAIMS

1. A solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters comprising the steps of:

- (a) reacting acylated carbohydrate with free fatty acid in the presence of an acid catalyst, under reduced pressure;
- (b) decolorizing and separating the reaction mixture obtained in step (a) into unreacted fatty acid layer and a carbohydrate fatty-ester layer;
- (c) precipitating out the unreacted acylated carbohydrate;
- (d) liberating the free hydroxyl groups by partial hydrolysis in the presence of an acid or base catalyst;
- (e) removing the unreacted free fatty acids and carbohydrate esters of low molecular-weight carboxylic acids during purification, and recycling the removed unreacted free fatty acids and carbohydrate esters to the starting reactant mixture

2. A solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters as set forth in Claim 1, wherein in step (a), no solvent is added thereto.

3. A solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters as set forth in Claim 1, wherein the unreacted acylated carbohydrate is





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precipitated out by cooling the carbohydrate fatty-ester layer to 10 to 0 degree C.

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4. A solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters as set forth in Claim 1, wherein step (a) is processed at a temperature ranging from 60 to 95 degree C.

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ABSTRACT OF THE DISCLOSURE

A SOLVENT-FREE TRANS-ACIDOLYSIS PROCESS FOR  
PREPARING EDIBLE SURFACE-ACTIVE CARBOHYDRATE  
FATTY-ACID ESTERS

5

The present invention relates to a low temperature, solvent-free trans-acidolysis process for preparing edible surface-active carbohydrate fatty-acid esters comprising the steps of:

- (a) reacting acylated carbohydrate with free fatty acid in the presence of an acid catalyst, under reduced pressure;
- (b) decolorizing and separating the reaction mixture obtained in step (a) into unreacted fatty acid layer and a carbohydrate fatty-ester layer;
- (c) precipitating out the unreacted acylated carbohydrate;
- (d) liberating the hydroxyl groups by partial hydrolysis in the presence of an acid or base catalyst;
- (e) removing the unreacted free fatty acids and carbohydrate esters of low molecular-weight carboxylic acids during purification, and recycling the removed unreacted free fatty acids and carbohydrate esters to the starting reactant mixture

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